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(54) **Process for obtaining pure lecithin which can be used directly for physiological purposes**

(57) Substantially pure lecithin is obtained from crude lecithin by extraction in an extraction stage using a gas under supercritical conditions of temperature and pressure. Various physiologically acceptable gases are mentioned. The gas is transferred to a separation stage where the temperature and/or pressure of the

gas is changed and extract separates out. The temperature and/or pressure of the gas are restored to the temperature and/or pressure required in the extraction stage and the gas is recycled thereto. Substantially pure lecithin is recovered from the extraction stage and extracted oily and other constituents are removed from the separation stage. Carbon dioxide is a preferred extracting gas and the process may be conducted continuously or batchwise.

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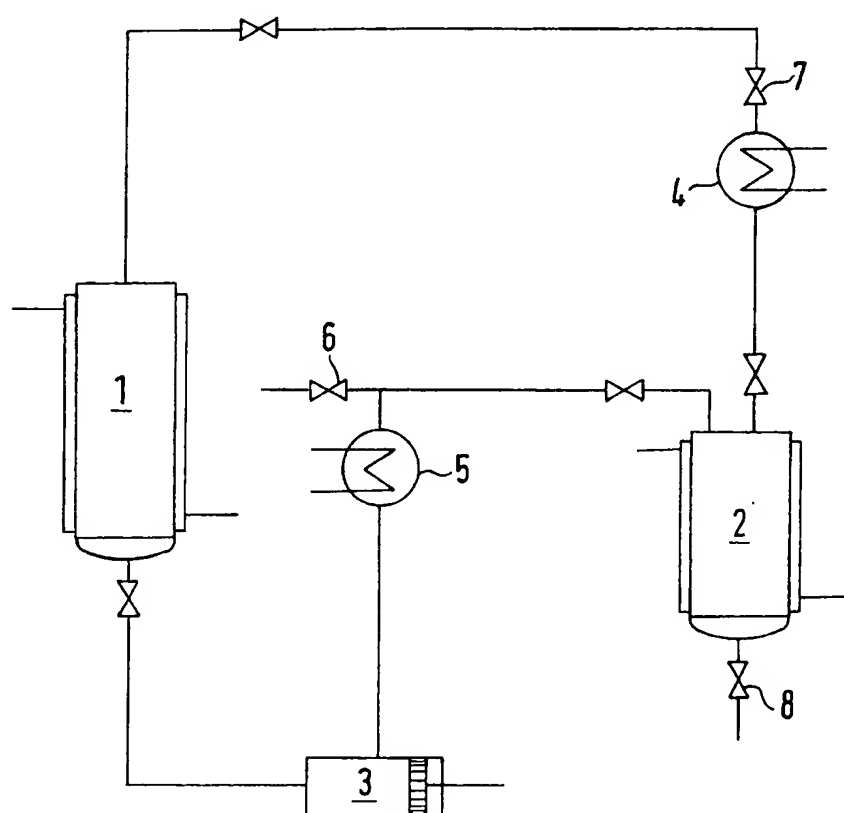
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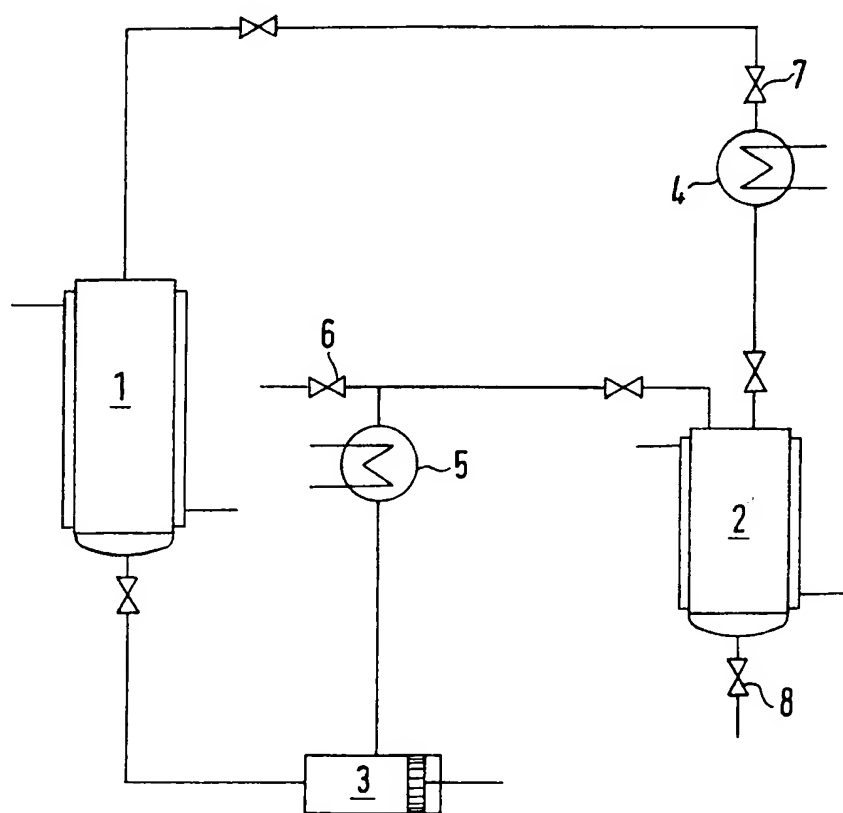
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SPECIFICATION

Process for obtaining pure lecithin which can be used directly for physiological purposes

This invention relates to a process for obtaining substantially pure lecithin, which can be used directly for physiological purposes.

The lecithins are understood to be a group of substances which belong to the phosphatide series. These substances, which contain the element phosphorus in the form of a phosphate ester, can be obtained by working up certain fat-containing vegetable and animal products.

The starting material for isolating, for example, soya-lecithin is soya beans. After cleaning, shelling and grinding the raw material, it is treated with solvents, whereby an oily product which contains the lecithin in colloidal solution is obtained by extraction. The lecithin content of the solution is increased further by an additional treatment with water at elevated temperature and by subsequent dehydration, a semi-solid, pasty material, that is to say crude lecithin, being obtained. This crude lecithin consists of about 60 to 70% of phosphatides and about 30 to 40% of oily constituents.

For further processing and industrial use of the lecithins, it is necessary to keep the oil content as low as possible. Since lecithins are widely used in the production, processing and preserving of foodstuffs and pharmaceutical and cosmetic preparations, lecithins are required to be of very high quality, especially with regard to their physiological acceptability.

It has been proposed to reduce the oil content further by expensive purification processes, in some cases by boiling out and steam stripping, or by further extraction with certain solvents and solvent mixtures. When these measures are taken, either a further, energy-intensive drying process, which is additionally subsequently carried out, is required, or, in the case of extraction, residual portions of the solvents used in large excess remain in the lecithin, so that the traces of foreign solvents must be removed. During this procedure, the lecithin is exposed to severe conditions, such as, for example, elevated temperature for prolonged periods, which increases the danger of the splitting of esters or of other decomposition processes.

The present invention is directed to a new process for obtaining pure lecithin which can be used directly for physiological purposes, by treating crude lecithin with a gas as an extracting agent, under conditions which are supercritical with respect to pressure and temperature. According to the present invention there is provided a process for obtaining from crude lecithin substantially pure lecithin which can be used directly for physiological purposes, comprising the steps of bringing the crude lecithin into contact with a gas, as an extracting agent, under conditions which are supercritical with respect to pressure and temperature in an extracting stage, transferring extract-containing

gas from the extraction stage to a separation stage, separating the extract-containing gas in the separation stage into gas and extract by changing the pressure and/or temperature, recycling the gas to the extraction stage, after restoring the gas to the pressure and/or temperature required in the extraction stage, and recovering substantially pure lecithin from the extraction stage.

Extraction processes which operate using supercritical gases as the extracting agent have already been proposed.

Thus, according to the process described in German Patent Specification No. 2,127,618, a hop extract is obtained by extraction by means of a supercritical gas. In this process, however, the resin constituents, essential oils and acid constituents are not removed in a selective manner to the same extent.

According to the process described in German Democratic Republic Patent Specification No. 41,362, for example, substances which are quite different in polarity, such as silicone oils, paraffin oils and aluminium sec-butyrate, can simultaneously be extracted side by side, using non-polar, super-critical gases.

According to the process described in German Offenlegungsschrift No. 2,709,033, camomile is extracted with a super-critical gas. In this process, relatively high proportions of undesired concomitant substances, such as, for example, lipoids, dyestuffs and the like, are obtained in the extract, in addition to the desired extracted substances; the content of these concomitant substances can indeed be reduced somewhat by a specific, expensive procedure, but the amount of lipoidal contents cannot be substantially reduced.

It was thus completely surprising and not to be expected, that it would be possible to separate the oily concomitant substances completely from the lipoidal lecithin by means of extraction with a supercritical gas.

In principle, any gas which is physiologically acceptable and which is chemically inert towards lecithin can be employed as the extracting agent, such as, for example, carbon dioxide, a halogenated hydrocarbon, such as one having the formula CHF_3 , $\text{CF}_3\text{—CF}_3$, CHCl=CF_2 , $\text{CF}_2\text{=CH}_2$, $\text{CF}_3\text{—CH}_3$, $\text{CHF}_2\text{—CH}_3$, CHF_2Cl , CF_3Cl , $\text{CF}_2\text{=CF}_2$, CF_2Cl_2 , $\text{CCl}_3\text{—CHCl}_2$ or C_3F_8 , sulphur hexafluoride, nitrous oxide, sulphur dioxide, a lower alkane or alkene, such as ethane, ethene, propane or propene or a mixture of two or more such gases. It is particularly preferred to use carbon dioxide as the extracting gas.

In the extraction stage, the substrate is exposed to the extracting agent under conditions under which the gas is supercritical both with respect to both temperature and pressure. The critical parameters of the gases mentioned above are known and available from the literature.

In the case where carbon dioxide is employed as the extracting agent, the extraction is carried out at a temperature in the range of from 31.3 to 100°C., preferably 35 to 80°C., and in particular 40 to 60°C., and under a pressure in the range of

SPECIFICATION

Process for obtaining pure lecithin which can be used directly for physiological purposes

This invention relates to a process for obtaining substantially pure lecithin, which can be used directly for physiological purposes.

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gas from the extraction stage to a separation stage, separating the extract-containing gas in the separation stage into gas and extract by changing the pressure and/or temperature, recycling the gas to the extraction stage, after restoring the gas to the pressure and/or temperature required in the extraction stage, and recovering substantially pure lecithin from the extraction stage.

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In the extraction stage, the substrate is exposed to the extracting agent under conditions under which the gas is supercritical both with respect to both temperature and pressure. The critical parameters of the gases mentioned above are known and available from the literature.

In the case where carbon dioxide is employed as the extracting agent, the extraction is carried out at a temperature in the range of from 31.3 to 100°C., preferably 35 to 80°C., and in particular 40 to 60°C., and under a pressure in the range of

from 72 to 800 bars, preferably 200 to 500 bars and in particular 300 to 400 bars.

In the case where an extracting agent other than carbon dioxide is employed, the lower and upper limits for the pressure and temperature for the extraction and separation are determined by the corresponding critical data.

After the extraction, the extracting agent which contains extract is transferred from the extraction stage to a separation stage in order to separate the extract from the extracting agent. Separation is thereby achieved by changing the temperature and/or pressure, it being preferable to change both parameters.

In the case where the temperature is changed, it is advantageous to reduce the temperature; the temperature is preferably reduced to below the critical temperature, thus, for carbon dioxide, for example, to a temperature in the range of from 10 to 32°C., in particular 15 to 25°C.

In the case where the pressure is changed, it is advantageous to reduce the pressure. The pressure is thus reduced to below the critical pressure, that is to say, for carbon dioxide, for example, to 10 to 72 bars, preferably 30 to 60 bars.

If the procedure described is followed, the change in temperature and/or pressure reduces the solvent power of the extracting agent for the extracted oily concomitant substances to such an extent that these separate out from the extracting agent in the separation stage and can be removed during the extraction, for example through a lock system, or when the extraction has ended.

The extracting agent can be re-used directly, without further labour-intensive and energy-intensive purification, so that, furthermore, no additional emission problems occur. In order for the extracting agent to be re-introduced into the extraction stage, it is only necessary to convert it into the supercritical state with respect to temperature and pressure, that is to say the change in temperature and/or pressure effected for the separation is reversed.

If the present process is carried out batchwise, the lecithin (pure lecithin) which has been purified from its concomitant substances can be removed from the extraction stage when the extraction has ended. If the process is carried out continuously, however, it is also possible to remove the pure lecithin — for example via a lock system — during the extraction process. In both types of operation, extraction times of 1 to 12 hours, preferably 3 to 7 hours, suffice to obtain the end product at a desired degree of purity.

The present process thus makes it possible to obtain the desired product in a short time while minimising the expense of apparatus, labour and energy, and while preventing pollution of the environment. In particular, the recirculation of the extracting agent makes it possible to automate the course of the process and hence to carry out the extraction in a particularly economical manner. The pure lecithin obtained can be passed directly to its use in foodstuffs and pharmaceutical or

cosmetic products.

In order to enable the invention to be more readily understood, reference will now be made to the accompanying drawing which illustrates diagrammatically, and by way of example, an embodiment thereof and which shows apparatus for extracting pure lecithin from a substrate comprising crude lecithin.

The apparatus shown in the drawing comprises an extraction vessel 1 and a separation vessel 2 linked in a closed circulating system for an extracting agent. The substrate (crude lecithin) which is to be extracted is first introduced into the extraction vessel 1. An extracting agent is introduced via an inlet valve 6 into the apparatus, it being unnecessary first to displace the air present in the apparatus. The extracting agent is compressed and delivered by a compressor 3 and brought to the particular temperature desired in the individual process stages by means of heat exchangers 4 and 5. The extraction vessel 1 and the separation vessel 2 are constructed as pressure vessels which can be thermostatically controlled, so that an exact adjustment of the temperature in the process stages carried out in these vessels is possible with the aid of an additional temperature control system.

After the pressures and temperatures desired in the individual stages have been reached, the inlet valve 6 is closed and the extracting agent is circulated. The extracting agent which has been brought to the extraction conditions by the heat exchanger 5 and the compressor 3 enters the extraction vessel 1 in intimate contact with the substrate. The extracting agent now containing the extract leaves this vessel via a screening device which is intended to prevent the entrainment of solid constituents into the separation vessel and a blockage of valves and the like, and the extracting agent is introduced via a throttle valve 7 and the heat exchanger 4 into the separation vessel 2 where the extract and the extracting agent separate. While the latter is recycled into the circulation via the heat exchanger 5 towards the compressor 3, the extract is removed, if appropriate during the extraction via a lock system at a take-off point 8, or directly from the separation vessel 2 after the extraction has ended; likewise the pure lecithin is removed from the extraction vessel 1.

If, instead of a single pressure vessel 1, two or more pressure vessels are used, the present process according to the invention can easily be carried out continuously. In this case, it is possible, in a manner which is in itself known, continuously to remove the pure lecithin from one vessel and to charge this vessel with fresh substrate, while the extraction is continued, without interruption, in the other pressure vessel or vessels of the extraction stage.

The individual vessels can be connected in parallel or in series. In the latter case, it is particularly advantageous to carry out the extraction "in countercurrent", that is to say for the particular pressure vessel containing the

from 72 to 800 bars, preferably 200 to 500 bars and in particular 300 to 400 bars.

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If the procedure described is followed, the change in temperature and/or pressure reduces the solvent power of the extracting agent for the extracted oily concomitant substances to such an extent that these separate out from the extracting agent in the separation stage and can be removed during the extraction, for example through a lock system, or when the extraction has ended.

The extracting agent can be re-used directly, without further labour-intensive and energy-intensive purification, so that, furthermore, no additional emission problems occur. In order for the extracting agent to be re-introduced into the extraction stage, it is only necessary to convert it into the supercritical state with respect to temperature and pressure, that is to say the change in temperature and/or pressure effected for the separation is reversed.

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The individual vessels can be connected in parallel or in series. In the latter case, it is particularly advantageous to carry out the extraction "in countercurrent", that is to say for the particular pressure vessel containing the

substrate which has been extracted to the greatest extent to be charged with fresh extracting agent coming directly from the compressor 3, and for the particular vessel containing the freshest substrate to be connected as the last member of the extraction stage.

More detailed process conditions are illustrated in the following Examples of the use of the apparatus shown in the drawing; the pressures and temperatures in the various vessels being designated as follows:—

Extraction vessel: Pressure: p_1
Temperature: t_1

Separation vessel: Pressure: p_2
Temperature: t_2

EXAMPLE 1

1000 g. of crude lecithin paste are introduced into the extraction vessel; the vessel is closed and the paste is extracted with carbon dioxide gas under supercritical conditions.

$p_1 = 400$ bars $p_2 = 50$ bars
 $t_1 = 60^\circ\text{C}$ $t_2 = 20^\circ\text{C}$

Extraction time: 4 hours

This gives 580 g. of a solid, light-yellow substance, that is to say pure lecithin, which is removed from the extraction vessel. 380 g. of yellow-coloured, clear oil and an aqueous phase weighing 30 g. which has settled out, are drained off from the separation vessel.

EXAMPLE 2

100 g. of crude lecithin paste with an oil content of about 30% are introduced into the extraction vessel; the vessel is closed and the paste is extracted with supercritical carbon dioxide.

$p_1 = 300$ bars $p_2 = 40$ bars
 $t_1 = 40^\circ\text{C}$ $t_2 = 20^\circ\text{C}$

Extraction time: 6 hours

69 g. of pure lecithin in the form of a solid light-yellow, non-hygroscopic substance remain in the extraction vessel. 31 g. of yellow-coloured, clear oil are found in the separation vessel.

When checked by thin layer chromatography, the products from the two examples prove to be free from oily components.

CLAIMS

1. A process for obtaining from crude lecithin substantially pure lecithin which can be used directly for physiological purposes, comprising the steps of bringing the crude lecithin into contact with a gas, as an extracting agent, under conditions which are supercritical with respect to

pressure and temperature in an extracting stage, transferring extract-containing gas from the extraction stage to a separation stage, separating the extract-containing gas in the separation stage into gas and extract by changing the pressure and/or temperature, recycling the gas to the extraction stage, after restoring the gas to the pressure and/or temperature required in the extraction stage, and recovering substantially pure lecithin from the extraction stage.

2. A process as claimed in claim 1, wherein the substantially pure lecithin is recovered continuously from the extraction stage.

3. A process as claimed in claim 1, wherein the substantially pure lecithin is recovered batch wise from the extraction stage.

4. A process as claimed in claim 1, wherein the gas is a physiologically acceptable gas.

5. A process as claimed in any one of claims 1 to 4, wherein the gas is selected from carbon dioxide, a halogenated hydrocarbon, sulphur hexafluoride, nitrous oxide, sulphur dioxide and lower alkanes and alkenes.

6. A process as claimed in claim 5, wherein the halogenated hydrocarbon is selected from CHF_3 , CF_3-CF_3 , $\text{CHCl}=\text{CF}_2$, $\text{CF}_2=\text{CH}_2$, CF_3-CH_3 , CHF_2-CH_3 , CHF_2Cl , CF_3Cl , $\text{CF}_2=\text{CF}_2$, CF_2Cl_2 , $\text{CCl}_3-\text{CHCl}_2$ and C_3F_8 .

7. A process as claimed in claim 5, wherein the lower alkane or alkene is selected from ethane, ethene, propane and propene.

8. A process as claimed in any one of claims 1 to 4, wherein the gas is carbon dioxide.

9. A process as claimed in any one of claims 1 to 8, wherein the extraction stage is operated under a pressure of from 72 to 800 bars.

10. A process as claimed in any one of claims 1 to 9, wherein the extraction stage is operated at a pressure of 200 to 500 bars.

11. A process as claimed in any one of claims 1 to 10, wherein the extraction stage is operated at a pressure of 300 to 400 bars.

12. A process as claimed in any one of claims 1 to 11, wherein the extraction stage is operated at a temperature of from 31.3 to 100°C .

13. A process as claimed in any one of claims 1 to 12, wherein the extraction stage is operated at a temperature of 35 to 80°C .

14. A process as claimed in any one of claims 1 to 12, wherein the extraction stage is operated at a temperature of 40 to 60°C .

15. A process as claimed in any one of claims 1 to 14, wherein the change in pressure is a reduction in pressure and the change in temperature is a reduction in temperature to subcritical conditions, both temperature and pressure being changed.

16. A process as claimed in any one of claims 1 to 15, wherein the separation stage is operated under a pressure of 10 to 72 bars.

17. A process as claimed in any one of claims 1 to 16, wherein the separation stage is operated under a pressure of 30 to 60 bars.

18. A process as claimed in any one of claims 1 to 17, wherein the separation stage is operated at

substrate which has been extracted to the greatest extent to be charged with fresh extracting agent coming directly from the compressor 3, and for the particular vessel containing the freshest substrate to be connected as the last member of the extraction stage.

More detailed process conditions are illustrated in the following Examples of the use of the apparatus shown in the drawing; the pressures and temperatures in the various vessels being designated as follows:—

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pressure and temperature in an extracting stage, transferring extract-containing gas from the extraction stage to a separation stage, separating the extract-containing gas in the separation stage into gas and extract by changing the pressure and/or temperature, recycling the gas to the extraction stage, after restoring the gas to the pressure and/or temperature required in the extraction stage, and recovering substantially pure lecithin from the extraction stage.

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3. A process as claimed in claim 1, wherein the substantially pure lecithin is recovered batch wise from the extraction stage.

4. A process as claimed in claim 1, wherein the gas is a physiologically acceptable gas.

5. A process as claimed in any one of claims 1 to 4, wherein the gas is selected from carbon dioxide, a halogenated hydrocarbon, sulphur hexafluoride, nitrous oxide, sulphur dioxide and lower alkanes and alkenes.

6. A process as claimed in claim 5, wherein the halogenated hydrocarbon is selected from CHF_3 , CF_3-CF_3 , $\text{CHCl}=\text{CF}_2$, $\text{CF}_2=\text{CH}_2$, CF_3-CH_3 , CHF_2-CH_3 , CHF_2Cl , CF_3Cl , $\text{CF}_2=\text{CF}_2$, CF_2Cl_2 , $\text{CCl}_3-\text{CHCl}_2$ and C_3F_8 .

7. A process as claimed in claim 5, wherein the lower alkane or alkene is selected from ethane, ethene, propane and propene.

8. A process as claimed in any one of claims 1 to 4, wherein the gas is carbon dioxide.

9. A process as claimed in any one of claims 1 to 8, wherein the extraction stage is operated under a pressure of from 72 to 800 bars.

10. A process as claimed in any one of claims 1 to 9, wherein the extraction stage is operated at a pressure of 200 to 500 bars.

11. A process as claimed in any one of claims 1 to 10, wherein the extraction stage is operated at a pressure of 300 to 400 bars.

12. A process as claimed in any one of claims 1 to 11, wherein the extraction stage is operated at a temperature of from 31.3 to 100°C .

13. A process as claimed in any one of claims 1 to 12, wherein the extraction stage is operated at a temperature of 35 to 80°C .

14. A process as claimed in any one of claims 1 to 12, wherein the extraction stage is operated at a temperature of 40 to 60°C .

15. A process as claimed in any one of claims 1 to 14, wherein the change in pressure is a reduction in pressure and the change in temperature is a reduction in temperature to subcritical conditions, both temperature and pressure being changed.

16. A process as claimed in any one of claims 1 to 15, wherein the separation stage is operated under a pressure of 10 to 72 bars.

17. A process as claimed in any one of claims 1 to 16, wherein the separation stage is operated under a pressure of 30 to 60 bars.

18. A process as claimed in any one of claims 1 to 17, wherein the separation stage is operated at

a temperature of 10 to 32°C.

19. A process as claimed in any one of claims 1 to 18, wherein the separation stage is operated at a temperature of 15 to 25°C.

5 20. A process as claimed in any one of claims 1 to 19, wherein the crude lecithin is extracted for 1 to 12 hours.

21. A process as claimed in any one of claims 1 to 20, wherein the crude lecithin is extracted for 3

10 to 7 hours.

22. A process for obtaining from crude lecithin substantially pure lecithin substantially as hereinbefore described with reference to the accompanying drawing and/or in either of the foregoing Examples.

15 23. Substantially pure lecithin obtained by the process claimed in any preceding claim.

a temperature of 10 to 32°C.

19. A process as claimed in any one of claims 1 to 18, wherein the separation stage is operated at a temperature of 15 to 25°C.

5 20. A process as claimed in any one of claims 1 to 19, wherein the crude lecithin is extracted for 1 to 12 hours.

21. A process as claimed in any one of claims 1 to 20, wherein the crude lecithin is extracted for 3

10 to 7 hours.

22. A process for obtaining from crude lecithin substantially pure lecithin substantially as hereinbefore described with reference to the accompanying drawing and/or in either of the foregoing Examples.

15 23. Substantially pure lecithin obtained by the process claimed in any preceding claim.